

PATENT SPECIFICATION

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(54) DETERGENT COMPOSITIONS

(71) We, COLGATE-PALMOLIVE COMPANY, a Corporation organised and existing under the Laws of the State of Delaware, United States of America, of 300, Park Avenue, New York, New York 10022, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to detergent compositions, particularly heavy duty built detergent compositions suitable for washing clothes.

Heavy duty built detergent compositions generally contain an organic detergent and a substantial proportion of a builder salt, usually present in amount equal to or greater than the amount of organic detergent. It is not unusual, especially in the washing of white or colour-fast garments or linen, for the housewife to add a hypochlorite bleach, usually sodium hypochlorite solution (i.e. conventional household chlorine bleach), to the wash water.

The present invention provides a heavy duty built detergent composition which yields a much whiter and stain-free wash, when used in the presence of household chlorine bleach, than conventional commercial heavy duty built detergent compositions.

According to the present invention a built detergent composition, suitable for use in the washing of clothes, comprises, as the detergent component, at least one organic detergent selected from anionic, cationic and nonionic organic detergents, a builder salt and from ¼% to 1% of alkali metal bromide (e.g. sodium bromide).

The amount of bromide preferably does not exceed ½%. An amount of ¼% to ½% is usually sufficient to give a good bleaching and sanitizing effect without substantial undesirable residual odour on the washed clothes

or damage thereto, when the composition is employed in conventional household washing machines at the usual concentration of about 0.15% in the wash water, together with about 90 ppm of chlorine (supplied by half a cup of the usual commercial aqueous 5¼% sodium hypochlorite solution). The effects attained with the built detergent compositions of this invention, when using half a cup of the commercial sodium hypochlorite solution, are superior to those attained with the best commercial built detergents in the presence of twice as much of the NaOCl solution.

The organic detergent is preferably of the anionically active type. Examples are water-soluble salts of sulphonyl-containing detergents such as a sulphonate or sulphate having a long hydrophobic chain (of e.g. 10—20 carbon atoms). For example, there may be used higher alkylbenzene sulphonates (preferably having 10—18 carbon atoms in the alkyl group, such as dodecyl or tridecyl benzene sulphonate); paraffin sulphonates; olefin sulphonates; (made, for example, by reacting highly diluted gaseous sulphur trioxide with an alpha-olefin followed by heating with alkali, and containing hydroxy alkanesulphonates and alkenyl sulphonates); ethoxylated fatty alcohol sulphonates (e.g. a sulphate of an ethoxylated higher alkanol having 1—5, preferably about 3, oxyethylene groups per molecule); sulphuric acid esters of polyhydric alcohols incompletely esterified with higher fatty acids, e.g. coconut oil monoglyceride monosulphate and tallow diglyceride monosulphate; long chain pure or mixed alkyl sulphates, e.g. lauryl sulphate and cetyl sulphate; fatty acid esters of hydroxy sulphonic acid, e.g. higher fatty acid esters of low molecular weight alkylol sulphonic acids, such as fatty acid esters of isothionic acid; fatty acid ethanolamide sulphonates; and fatty acid amides of amino alkyl sulphonic acids, e.g. lauric acid amide of

[Price 25p]

taurine. Other suitable synthetic anionic detergents are water-soluble soaps of higher fatty acids such as the sodium soap of a 75:25 mixture of tallow and coconut oil fatty acids.

5 Although the anionic detergents are preferred, cationic and nonionic detergents may be also employed in whole or as part of the detergent, provided they are compatible with the other components of the composition under conditions of storage and use thereof.

10 As cationic detergents there may be mentioned the long chain alkyl quaternary ammonium compounds, e.g. cetyl quaternary ammonium salts. Within this class are cetyl trimethyl ammonium chloride and cetyl pyridinium chloride. Another cationic agent is diethylene amino ethyl oleyl amide.

15 The non-ionic agents are also well known in the art including polyoxyethylene ethers of alkyl aromatic hydroxy bodies (e.g. alkylated polyoxyethylene phenols), polyoxyethylene ethers of long chain aliphatic alcohols, and polyoxyethylene ethers of hydrophobic propylene oxide polymers, e.g. the condensate of ethylene oxide with polypropylene glycol, which condensate contains 80% ethylene oxide and has a molecular weight of about 1700, and isooctyl phenoxy polyoxyethylene ethanol having about 8.5 ethanoxyl groups per molecule. Alkyl amine oxide detergents such as lauryl or myristyl dimethyl amine oxides may be used.

20 The anionic and cationic surface active agents are commonly used in the form of their water-soluble salts. For the synthetic anionic compounds, the alkali metal (e.g. sodium, potassium) salts are preferred; though other salts such as ammonium, amine, alkylolamine and alkaline earth metals (e.g. calcium and magnesium) salts may be used if desired. For the cationic agents the chloride, sulphate, acetate and other salts may be employed satisfactorily.

25 When an alkyl benzene sulphonate detergent is used, its alkyl groups may be essentially linear (and biodegradable) or branched chain (e.g. derived from propylene tetramer) and the alkyl-benzene sulphonate may be a blend containing various alkyl chain lengths. When linear alkyl groups are present, the benzene ring is preferably attached in large part at the 3 or higher (e.g. 4, 5, 6 or 7) position of the alkyl group and the content of isomers in which the benzene ring is attached at the 2 or 1 position is correspondingly low (e.g. below 50%). Particularly preferred materials are set forth in British Patent Specification No. 1,087,337.

30 The builder salt is preferably a condensed phosphate alkali metal salt such as a polyphosphate or pyrophosphate, e.g. tetrasodium pyrophosphate, pentasodium tripolyphosphate, (which may be present in either of its two common "Forms", or in a mixture of Forms, but preferably Form II), sodium hexameta-

phosphate or the corresponding potassium salts, alone or in a mixture with each other. For solid compositions pentasodium tripolyphosphate is especially suitable; for liquid compositions, tetrapotassium pyrophosphate is particularly preferred. Other builders may be of a type well known in the art; which may include alkali metal, alkaline earth metal, ammonium and various ethanolanmonium salts, alone or in combination, the preferred builders being sodium and potassium salts. Examples are water-soluble phosphates, silicates, carbonates, bicarbonates, borates, sulphates and chlorides as well as salts of organic acids such as ethylene diamine tetraacetic acid or nitrilotriacetic acid. Combinations of a salt of nitrilotriacetic acid (e.g. the trisodium salt) with the condensed phosphate (e.g. in ratio about two parts of the former to three to six parts of the phosphate) are particularly effective.

35 The detergent composition generally contains from 5% to 45% (on a solids basis) of organic synthetic detergent. The amount of builder salt is generally at least equal to two thirds the amount of organic detergent. Preferred solid detergent compositions contain from 10% to 20% of the organic detergent and, usually, in the range from two to eight, (preferably from two to five) parts of builder salt per part of detergent. Preferably the proportion and character of the builder salt are such that a 0.15% solution of the built detergent composition in water has a pH of about 9½ to 11.

40 When the composition is to be used for washing fabrics containing nylon which, as is well known, tend to become yellowed on aging, it is desirable to include bleach-stable brightening agents and colouring agents in the built detergent composition. Particularly suitable brightening agents of this type are the known triazole stilbene brightening agents, such as the naphthotriazole stilbene mono- di- or poly-sulphonates having the sulphonate substitution vicinal to the ethylenic bridge or on any of the positions of the naphthalene ring. The amount of such brightener may be, for instance, 0.05% to 0.1% or more.

45 Other ingredients which may be present in the detergent composition are ingredients to give an additional germicidal effect such as halogenated carbanilides, e.g. trichlorocarbanilide, halogenated salicylanilide, e.g. tribromosalicylanilide, halogenated bis-phenols, e.g. hexachlorophene, halogenated trifluoromethyldiphenyl urea, and zinc salt of 1-hydroxy - 2 - pyridinethione (e.g. in amounts in the range from 0.01% to 5%); soil-suspending agents such as sodium carboxymethyl cellulose or polyvinyl alcohol, or a combination of these two, or other suitable water-soluble polymeric materials (the amount of suspending agent being, for example, in the range from 0.1% to 2%); other brighteners, foam boosters, e.g. mono- or di-

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ethanolamides of long chain fatty acids, such as the monoethanolamide of coconut oil fatty acids or of a 70:30 lauric-myristic acid blend (e.g. present in proportion of from $\frac{1}{2}$ % to 3%).

As previously indicated, the compositions of this invention are highly useful for washing clothes in the presence of sodium hypochlorite solution. In place of sodium hypochlorite, other sources of hypochlorite chlorine (i.e. chlorine present in hypochlorous ion, OCl^-) may be added to the wash water. These may be, for instance, water-soluble dry solid materials which generate hypochlorite ion on contact with, or dissolution in, water. Examples thereof are dry, particulate heterocyclic N-chloro imides such as trichloroisocyanuric acid, and dichloroisocyanuric acid and salts thereof such as sodium dichloroisocyanurate and potassium dichloroisocyanurate. Other imides may also be used such as N-chlorosuccinimide, N-chloromalonimide, N-chlorophthalimide and N-chloronaphthalimide. Additional suitable imides are the hydantoin salts such as 1,2-dichloro 5,5 dimethyl hydantoin; N - monochloro - 5,5 - dimethyl - hydantoin; methylenebis (N chloro - 5,5 - dimethylhydantoin); and 1,3 - dichloro - 5 - methyl - t - n - amylhydantoin. Other useful hypochlorite-liberating agents are trichloromelamine, N,N-dichlorobenzoylene urea, N,N - dichloro - p - toluene - sulphonamide and dry, particulate, water-soluble anhydrous inorganic salts such as lithium hypochlorite and calcium hypochlorite.

The following examples illustrate the invention. In these Examples and elsewhere in the specification all parts and percentages are by weight unless otherwise indicated.

EXAMPLE 1

A spray-dried detergent composition had the following approximate composition: 10% sodium linear tridecylbenzenesulphonate; 2% of the ethoxylation products made from ethylene oxide and primary alkanols of C_{14} — C_{15} chain length, the ethoxylation products containing 11 mols of oxyethylene per mol of alkanol; 2% of sodium soap of a mixture of 3 parts of tallow fatty acids and 1 part of coconut oil fatty acids; 34% of sodium sulphate; 34% of phosphate solids; 7% of sodium silicate solids ($\text{Na}_2\text{O}:\text{SiO}_2$ mol ratio 1:2.35); 0.25% of sodium bromide; 0.15% of ultramarine blue; 0.5% of sodium carboxymethyl cellulose; 0.2% of water-soluble polyvinyl alcohol; 0.44% of fluorescent brighteners; 0.09% of Phthalocyanine Blue WD; 0.01% of a phenolic antioxidant ("Iphol") and the balance moisture.

The composition was prepared by separately spray drying a light blue formulation and a dark blue formulation and mixing the resulting two spray dried powders in the proportions of 95:5 (light blue: dark blue), to give

a detergent formulation having a speckled appearance.

The light blue powder was made by spraying an aqueous slurry containing the ingredients described above (without the Phthalocyanine Blue); the phosphate in the composition was supplied by the addition of anhydrous pentasodium tripolyphosphate to the hot (165°F) aqueous mixture just before spraying the resulting slurry. The dark blue powder was made by spraying a mixture containing 96.7% of this slurry and 3.3% of an aqueous 30% dispersion of the Phthalocyanine Blue WD. The solids content of each slurry before spraying was about 60% (i.e. slurry had a total moisture content of about 40%).

The spraying was carried out in a spray tower into which the hot slurry was sprayed into a counter current stream of hot air. On entering the tower the hot air had a temperature of about 700°F and was at substantially atmospheric pressure, while the mixture being sprayed had a temperature of 165°F . The spray-dried particles were hollow beads, some of which adhered together in small clumps.

The detergent composition was a stable mixture which was highly effective for the bleaching and washing of clothes in an automatic washing machine at a concentration of 0.15% in wash water containing half a cup of commercial sodium hypochlorite (providing 90 ppm available chlorine) at a temperature of 120°F .

EXAMPLE 2

Another spray-dried detergent composition (having a greater bleach-promoting effect than the composition of Example 1) had the following approximate composition: 15 parts sodium linear tridecylbenzenesulphonate; 25.5 parts sodium sulphate; 34 parts phosphate solids; 7 parts sodium silicate solids ($\text{Na}_2\text{O}:\text{SiO}_2$ mol ratio 1:2.35); 5 parts trisodium nitrilotriacetate; 0.5 part sodium bromide; 0.1 part ultramarine blue; 0.003 part green dye (D & C Green No. 8); 0.09 part Phthalocyanine Blue WD; 0.5 part sodium carboxymethyl cellulose; 0.2 part water-soluble polyvinyl alcohol; 0.5 part fluorescent brighteners; 0.01 part phenolic antioxidant ("Iphol"); and 10 parts moisture.

The composition was prepared by separately spray drying a green formulation and a dark blue formulation and mixing the resulting two spray dried powders in the proportions of 95:5 (green:dark blue), to give a detergent formulation having a speckled appearance.

The green powder was made by spraying a slurry of the ingredients described above (without the Phthalocyanine Blue); the phosphate in the composition was supplied by the addition of anhydrous pentasodium tripolyphosphate to the hot (165°F) aqueous mixture just before spraying the resulting slurry.

The dark blue powder was made by spraying a mixture containing 96.7% of this slurry and 3.3% of an aqueous 30% dispersion of the Phthalocyanine Blue WD. The solids content of each slurry before spraying was about 60%.

The spraying was carried out in the same manner as described in Example 1.

The detergent composition was a stable mixture which was highly effective for the bleaching and washing of clothes in an automatic washing machine at a concentration of 0.15% in wash water containing half a cup of commercial sodium hypochlorite (providing 90 ppm available chlorine) at a temperature of 120°F.

In both Examples 1 and 2 the brighteners included (a) a naphthotriazole stilbene sulphonate brightener (Geigy "Tinopal RBS—200%," TINOPAL is a trade mark), (b) another stilbene brightener, bis (anilino diethanolamino triazinyl) stilbene disulphonic acid (c) another stilbene brightener, sodium bis (anilino morpholino triazinyl) stilbene disulphonate, and (d) an oxazole brightener, having a 1 - phenyl 2 - benzoxazole ethylene structure. The proportions of these in Example 1 were (a) 0.071%, (b) 0.071%, (c) 0.21% and (d) 0.088%. The proportions of these in Example 2 were (a) 0.09%, (b) 0.07%, (c) 0.25% and (d) 0.09%.

EXAMPLE 3

Example 2 was repeated except that in place of two thirds of the alkylbenzenesulphonate there was used an olefin sulphonate of 15 to 18 carbon atoms (made by reaction of the corresponding alpha-olefin and highly diluted sulphur trioxide followed by neutralization with aqueous sodium hydroxide and heat treatment, to produce a mixture of sodium alkenyl sulphonates and the corresponding sodium hydroxyalkanesulphonates).

EXAMPLE 4

A heavy duty liquid detergent formulation contained 8.75% sodium linear tridecylbenzene sulphonate; 10% sodium salt of the sulphate of an ethoxylation product made from 3 mols ethylene oxide and one mol primary C_{14} — C_{15} alkanol; 8.2% potassium xylene sulphonate; 1% lauric-myristic diethanolamide; 0.25% sodium carboxymethyl cellulose; 0.25% polyvinyl alcohol; 0.6% hydrogenated castor oil; 0.16% bis (anilino diethanolamino triazinyl) stilbene disulphonate (brightener); 0.06% naphthotriazole stilbene sulphonate brightener (Geigy "Tinopal RBS 200%"); 15% tetrapotassium pyrophosphate; 1.8% sodium sulphate; 0.25% sodium bromide; balance water with a small amount of perfume.

While the preferred alkali metal bromide is sodium bromide, other alkali metal bromides (e.g. potassium bromide or lithium bromide)

may be used in place of all or a part (e.g. 1/3) of the sodium bromide.

The composition of this invention may be used for washing fabrics containing natural fibres such as cotton and linen, or blends of such fibres (e.g. durable press cotton-polyester blends). They may be used for washing clothes in cool water (e.g. 40°—80° or 100°F) as well as in warm or hot water.

The invention also embraces a process for washing or laundering fabric articles in water, which process comprises dissolving in water a built detergent composition as described above to form a washing solution, separately incorporating into the water a bleach supplying hypochlorous ions, washing the articles in the resulting solution, and recovering the articles in effectively laundered and bleached condition from the solution.

Attention is invited to our British Patent Application No. 56276/70 (Serial No. 1238692) divided herefrom and which is directed to a non-liquid, built detergent composition, suitable for use in the presence of aqueous hypochlorite, comprising an organic detergent, a builder salt and from 1/4% to 1% of alkali metal bromide.

WHAT WE CLAIM IS:—

1. A built detergent composition, suitable for use in the washing of clothes in the presence of aqueous hypochlorite, comprising, as the detergent component, at least one organic detergent selected from anionic, cationic and nonionic organic detergents, a builder salt and from 1/4% to 1% of alkali metal bromide.

2. A composition as claimed in Claim 1 in which the detergent comprises a sulphonate detergent.

3. A composition as claimed in Claim 1 or Claim 2 in which the builder salt comprises a condensed phosphate.

4. A composition as claimed in any of Claims 1 to 3 in which the builder salt is a mixture of nitrilotriacetate builder salt and a condensed phosphate builder salt.

5. A composition as claimed in any of Claims 1 to 4 containing at least 2/3 part of builder salt per part of organic detergent.

6. A composition as claimed in any of Claims 1 to 5 which is dry and granular and has from 10 to 20% of organic detergent, from 2 to 8 parts of builder salt per part of detergent, and from 1/4% to 1/2% of sodium bromide.

7. A composition as claimed in any of Claims 1 to 3 in which the detergent is a sodium linear alkylbenzenesulphonate having 12 or 13 carbon atoms in the alkyl group and the builder salt is pentasodium tripolyphosphate, the composition including a bleach-stable naphthotriazole stilbene sulphonate brightener.

8. A built detergent composition substantially as described in any of the Examples.

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The dark blue powder was made by spraying a mixture containing 96.7% of this slurry and 3.3% of an aqueous 30% dispersion of the Phthalocyanine Blue WD. The solids content of each slurry before spraying was about 60%.

The spraying was carried out in the same manner as described in Example 1.

The detergent composition was a stable mixture which was highly effective for the bleaching and washing of clothes in an automatic washing machine at a concentration of 0.15% in wash water containing half a cup of commercial sodium hypochlorite (providing 90 ppm available chlorine) at a temperature of 120°F.

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EXAMPLE 3

Example 2 was repeated except that in place of two thirds of the alkylbenzenesulphonate there was used an olefin sulphonate of 15 to 18 carbon atoms (made by reaction of the corresponding alpha-olefin and highly diluted sulphur trioxide followed by neutralization with aqueous sodium hydroxide and heat treatment, to produce a mixture of sodium alkenyl sulphonates and the corresponding sodium hydroxyalkanesulphonates).

EXAMPLE 4

A heavy duty liquid detergent formulation contained 3.75% sodium linear tridecylbenzene sulphonate; 10% sodium salt of the sulphate of an ethoxylation product made from 3 mols ethylene oxide and one mol primary C_{11} — C_{13} alkanol; 8.2% potassium xylene sulphonate; 1% lauric-myristic diethanolamide; 0.25% sodium carboxymethyl cellulose; 0.25% polyvinyl alcohol; 0.6% hydrogenated castor oil; 0.16% bis (anilino diethanolamino triazinyl) stilbene disulphonate (brightener); 0.06% naphthotriazole stilbene sulphonate brightener (Geigy "Tinopal RBS 200%"); 15% tetrapotassium pyrophosphate; 1.8% sodium sulphate; 0.25% sodium bromide; balance water with a small amount of perfume.

While the preferred alkali metal bromide is sodium bromide, other alkali metal bromides (e.g. potassium bromide or lithium bromide)

may be used in place of all or a part (e.g. 1/3) of the sodium bromide.

The composition of this invention may be used for washing fabrics containing natural fibres such as cotton and linen, or blends of such fibres (e.g. durable press cotton-polyester blends). They may be used for washing clothes in cool water (e.g. 40°—80° or 100°F) as well as in warm or hot water.

The invention also embraces a process for washing or laundering fabric articles in water, which process comprises dissolving in water a built detergent composition as described above to form a washing solution, separately incorporating into the water a bleach supplying hypochlorous ions, washing the articles in the resulting solution, and recovering the articles in effectively laundered and bleached condition from the solution.

Attention is invited to our British Patent Application No. 56276/70 (Serial No. 1238692) divided herefrom and which is directed to a non-liquid, built detergent composition, suitable for use in the presence of aqueous hypochlorite, comprising an organic detergent, a builder salt and from 1/4% to 1% of alkali metal bromide.

WHAT WE CLAIM IS:—

1. A built detergent composition, suitable for use in the washing of clothes in the presence of aqueous hypochlorite, comprising, as the detergent component, at least one organic detergent selected from anionic, cationic and nonionic organic detergents, a builder salt and from 1/4% to 1% of alkali metal bromide.

2. A composition as claimed in Claim 1 in which the detergent comprises a sulphonate detergent.

3. A composition as claimed in Claim 1 or Claim 2 in which the builder salt comprises a condensed phosphate.

4. A composition as claimed in any of Claims 1 to 3 in which the builder salt is a mixture of nitrilotriacetate builder salt and a condensed phosphate builder salt.

5. A composition as claimed in any of Claims 1 to 4 containing at least 2/3 part of builder salt per part of organic detergent.

6. A composition as claimed in any of Claims 1 to 5 which is dry and granular and has from 10 to 20% of organic detergent, from 2 to 8 parts of builder salt per part of detergent, and from 1/4% to 1% of sodium bromide.

7. A composition as claimed in any of Claims 1 to 3 in which the detergent is a sodium linear alkylbenzenesulphonate having 12 or 13 carbon atoms in the alkyl group and the builder salt is pentasodium tripolyphosphate, the composition including a bleachable naphthotriazole stilbene sulphonate brightener.

8. A built detergent composition substantially as described in any of the Examples.



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9. A process for washing or laundering fabric articles in water which comprises dissolving a detergent composition as claimed in any of Claims 1 to 8 in water to form a washing solution, separately incorporating into the water a bleach supplying hypochlorous ion, washing the articles in the resulting solu-

tion, and recovering the articles in effectively laundered and bleached condition from the solution.

KILBURN & STRODE,
Chartered Patent Agents,
Agents for the Applicants.

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